

Effect of Ascorbic Acid Concentration on the Stability of Tartrate-Capped Silver Nanoparticles

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Abstract: In this work, tartrate-capped silver nanoparticles (AgNPs) by reducing Ag^+ ions into Ag^0 using L-ascorbic acid and capping disodium tartrate have been prepared. The reaction was carried out at room temperature in an alkaline medium of pH 11 to obtain a rapid and one-step green synthesis method. The effect of L-ascorbic acid concentration on the synthesis preparation was studied to investigate their impact on the particle size, morphology, and stability of the AgNPs. The obtained tartrate capped AgNPs have SPR absorbance in 390–410 nm. They have a spherical shape, as confirmed by TEM. Increasing L-ascorbic acid concentrations from 25 mM to 100 and 200 mM leads to the 27, 17, and 11 nm particle size distributions. They give the zeta potential of -33.5, -20.8, and -21.3, respectively. After a week, the decreasing absorbance peaks were 0.151, 0.0105, and 0.336 a.u. The optimum L-ascorbic acid concentration was obtained at 100 mM, indicated by the smallest FWHM point. Thus, we may conclude that lower or higher levels of reducing agents resulted in low stability. Therefore, controlling L-ascorbic acid concentration is an important parameter. A sufficient concentration and an appropriate capping agent can produce good nanoparticle stability essential for further application.

Keywords: silver nanoparticles; L-ascorbic acid; disodium tartrate; green synthesis; stability

INTRODUCTION

Metal nanoparticles display unique physicochemical properties different from bulk metal, including large surface area, high-volume ratio, powerful optical, conductivity, catalytic, and magnetic properties [1]. Their properties are promising for various purposes, such as biological [2-3], chemical [4-5], and environmental [6-7] applications. AgNPs showed better performance in many applications, especially for sensing, than AuNPs because of the higher extinction coefficient, the ratio of scattering to extinction, field enhancements, and extinction bands, which are powerful in many applications, especially for sensing [8]. However, the lower chemical stability of silver nanoparticles becomes weakness of their properties, but many studies have been conducted to improve their stability by using a capping agent [8]. As a consequence, silver nanoparticles are popular for many purposes. These physicochemical nanostructure properties could be

maintained by understanding that stability is an essential parameter for fundamental and applied studies explained as a function of composition, size, shape, and surface chemistry [9-10].

Recently, green chemistry topics have rapidly grown in various research fields. Green synthesis design related to green chemical reactants, processes, and products reduced the use of hazardous materials and has become a critical issue in the nanomaterial topics [1]. Various synthesis strategies, including chemical, physical, and biological methods, have been developed to produce AgNPs [11]. The chemical reduction method achieved by reducing a metal salt is the most frequently chosen among other methods because easy to control the reaction process and product [12]. Factors that affect the reduction process include the solvent medium, reducing, and capping agent [1]. Previous research reported using organic solvents and environmentally hazardous reducing

agents in the wet chemical reduction methods (i.e., hydrazine, sodium borohydride, dimethyl-formamide, formaldehyde, sodium hypophosphite, or hydroxylamine hydrochloride) [1]. L-ascorbic acid has been applied previously for green nanoparticles synthesized [11,13-14]. It is a green chemical that is a highly water-soluble compound, biodegradable, and environmentally friendly. It is an effective reducing agent to mediate the formation of spherical silver nanoparticles than other reducing agents [15]. Besides these advantages, the low stability of silver nanoparticles using L-ascorbic acid as a reducing agent has seldom been reported. The zeta potential is one of the parameters that indicate the stability of metal nanoparticles. The stable metal nanoparticles have a zeta potential value $> +25$ mV or < -25 mV [13]. Previous research synthesized PVA capped AgNPs using L-ascorbic acid as a reducing agent, resulting in the zeta potential of -10.40 mV [16] and L-ascorbic acid capped AuNPs zeta potential of -4 mV [17]. The weakness related to the properties of L-ascorbic acid is that it is easy to degrade. It decomposes into biologically inactive species in the air and oxygen, at high temperatures and in moist conditions [14]. The shortcomings of L-ascorbic acid can be overcome by adding several capping agents to reaction media to maintain the stability of metal nanoparticles. We used disodium tartrate as the capping agent that acts as stabilizing agents and ligands in an aqueous solution. Tartrate ligand is biocompatible, highly soluble in water, and environmentally benign. Meanwhile, it shows optical properties such as intrinsic fluorescence and enhanced photocatalytic activities [18]. The tartrate ion consists of the carboxyl group, tetrahedral carbon, and hydroxyl oxygen atom. In addition, some tartrate molecules are hydrated, i.e., they contain one, two, three, or four molecules of water [19].

The size and morphology of synthesized metal nanoparticles are essential to study because they determine the application's performance. The synthesis and catalytic properties of spherical Cu NPs were reported using L-ascorbic acid as a reducing and capping agent. The result indicates that the size of Cu NPs decreases (28 nm to 12 nm), and the catalytic activity increases along with the concentration of L-ascorbic acid [20]. An

increasing concentration of L-ascorbic acid is produced in sharp Au NPs called Au-sea urchin-like architecture. The particle size distribution was 600–800 nm which is made up of some nanopricks with an average length of 100–200 nm. SERS studies show that its performance is superior [21]. Synthesized Au NPs were done using sodium tartrate as a mild reducing agent. The result AuNPs showed that the increasing sodium tartrate concentration decreased particle size [22]. The study about the influence of tartaric acid concentration on structural and optical properties of Cu Se nanoparticles revealed that the particle size decreases with an increase in tartaric acid concentration, and the hexagonal structure was seen. The prepared Cu Se NPs' bandgap increased [23]. Ascorbic acid capped AgNPs [24], and tartrate capped AgNPs [25] were also chosen as capping for selective and sensitive Cr ion detection using the hydroxyl group as the conjugating site.

This study aimed to investigate the effect of L-ascorbic acid concentrations on the stability and properties of tartrate capped silver nanoparticles during storage. For this purpose, we prepared the tartrate capped silver nanoparticles using various concentrations of L-ascorbic acid as a reducing agent and capping with disodium tartrate. Then, we characterized the produced tartrate capped AgNPs in terms of particle size, morphology, and stability, important for further applications.

■ EXPERIMENTAL SECTION

Materials

Silver nitrate (AgNO_3) MERCK 1.01512, L-ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) MERCK 1.00468, disodium tartrate dihydrate ($\text{C}_4\text{H}_8\text{Na}_2\text{O}_8 \cdot \text{xH}_2\text{O}$) MERCK 1.06663, and sodium hydroxide (NaOH) MERCK 1.06498 in the analytical grade were used without further purification.

Instrumentation

UV-Visible spectra were collected on a SPECORD 200 PLUS Analytic Jena UV-Visible spectrophotometer. TEM images were taken on a JEOL JEM-1400 electron microscope operated at an accelerating voltage of 120 kV, in which the colloid was dropped onto the

copper grid. The distribution of AgNPs particle size was calculated by using ImageJ software. Zeta potential was obtained on HORIBA Scientific SZ-100.

Procedure

Silver nanoparticles were prepared with a reducing agent of L-ascorbic acid and a capping agent of disodium tartrate. Typically, a 1 mL L-ascorbic acid solution with various concentrations (25, 50, 75, 100, 150, and 200 mM) was added to 16.0 mL disodium tartrate 4 mM. It was adjusted to pH 11.0 by adding 0.6 mol L⁻¹ NaOH solution. The 200 μ L of the aqueous solution of AgNO₃ 0.01 M was added under a stirring speed of 600 rpm at room temperature for 60 min. The effects of L-ascorbic acid concentrations on the synthesis preparation of silver nanoparticles were studied. In addition, the influence of storage time on the nanoparticle size and properties was investigated to know the stability of the produced nanoparticles.

■ RESULTS AND DISCUSSION

UV-Visible Study of the Effect of Ascorbic Acid on the Synthesized Tartrate Capped AgNPs

The increasing concentration of L-ascorbic acid on the UV-Vis spectra data of produced tartrate capped AgNPs was presented in Fig. 1(a). The peak absorbances are seen at 390, 400, 394, 391, 398, and 393, with the full width at half-maximum (FWHM) of 182.94, 147.01,

121.22, 108.65, 152.36, and 167.58 nm, respectively. The SPR absorbance of AgNPs appears at 350–450 nm [26]. The full width at half maxima (FWHM) of the SPR peaks describes the homogeneity of the nanoparticle size distribution. Increasing the FWHM values is proportional to increased particle size or polydispersity [27]. The UV-Vis spectra data concluded that the optimum L-ascorbic acid concentration was obtained at 100 mM, with the narrowest peak shape indicated by the smallest FWHM point. This result was confirmed by TEM in Fig. 2.

The resulted UV-Vis spectra of time evolution are shown in Fig. 1(b). The reducing and capping reaction was finished after 30 min as the UV-Vis absorption curves of 30–120 min overlapped. The L-ascorbic acid acts as the catalyst of the reaction. Therefore, a rapid synthesis can be carried out at room temperature without specific conditions. Insufficient or excess L-ascorbic acid concentration results in the broadening absorbance peak of the produce tartrate capped AgNPs.

On the other hand, low L-ascorbic acid concentration caused the silver salt to remain unchanged because of the insufficient reducing ability and generated the bigger size and irregular shape of NPs [28], as confirmed by TEM in Fig. 2(a). Meanwhile, the excess concentration resulted in explosive nucleation. This finding is related to the previous study that excessive concentration of caffeic acid [28] and ascorbic acid [21]

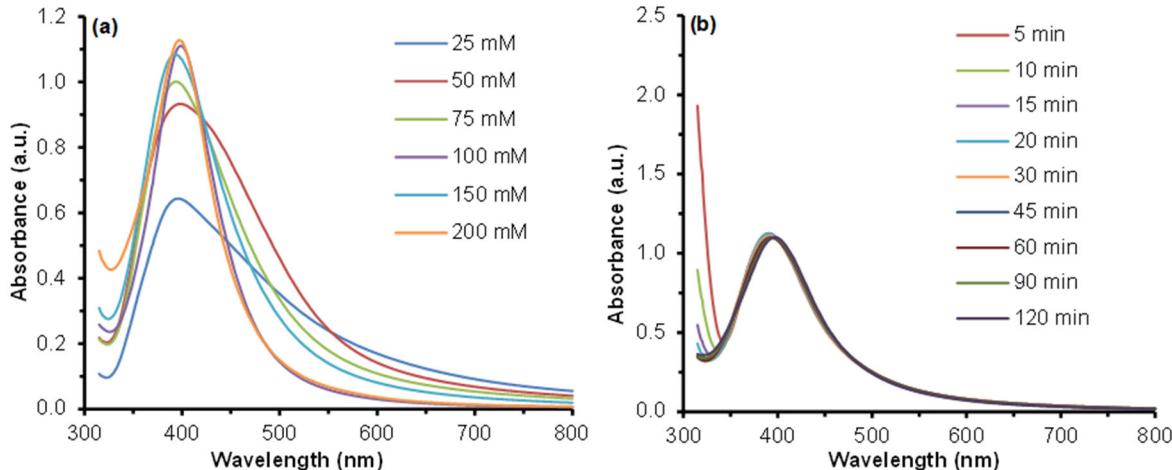


Fig 1. The UV-Visible spectrum of tartrate capped AgNPs (a) with various L-ascorbic acid concentrations, and (b) The synthesis time evolution

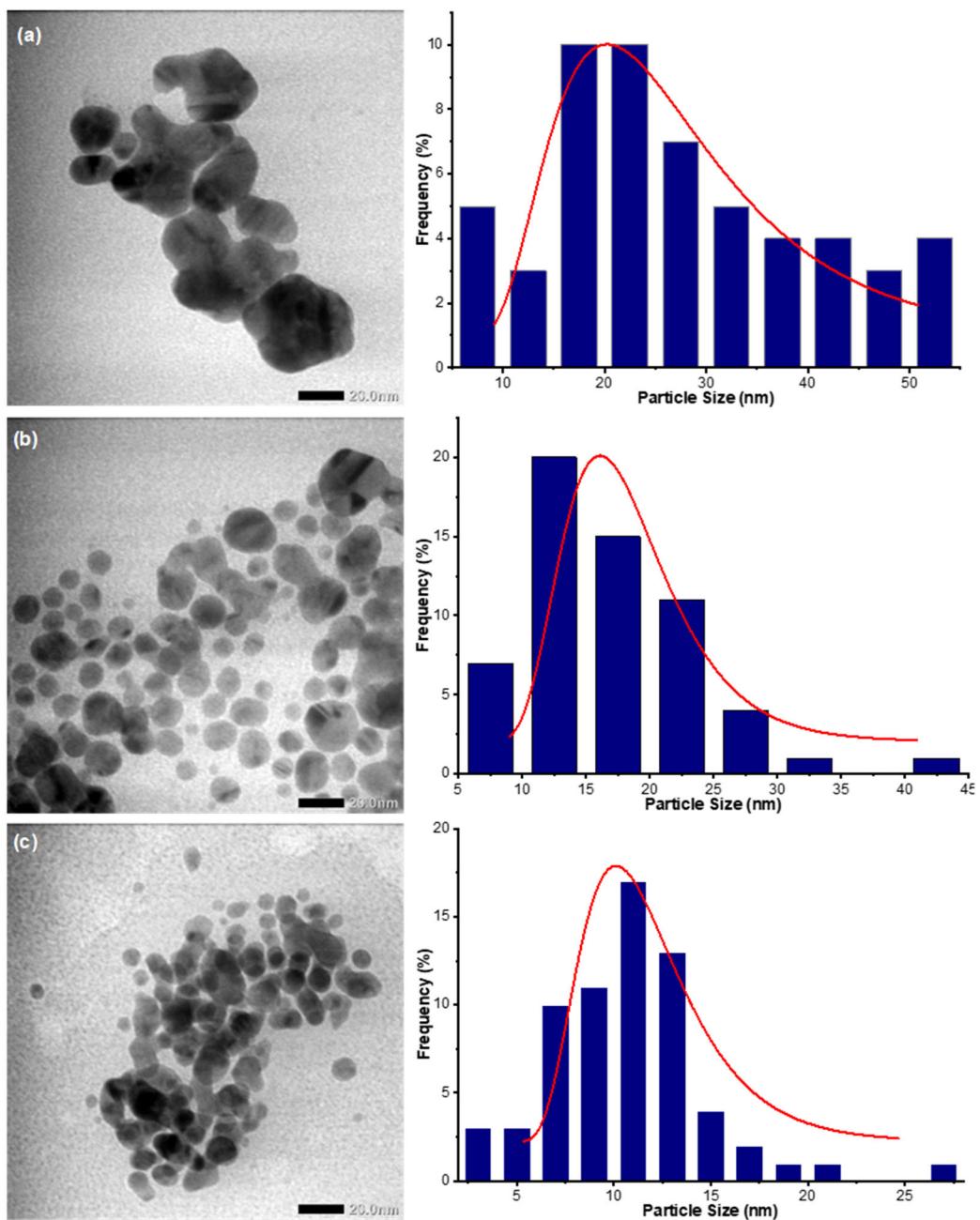


Fig 2. TEM images and particle size distribution histogram of the produced tartrate capped AgNPs with increasing concentration of L-ascorbic acid (a) 25, (b) 100, and (c) 200 mM with average particle sizes were 27, 17, and 11 nm

as reducing agents on the AuNPs synthesis generated sea-urchin architecture Au particle because of the rapid nucleation.

Metal nanoparticles are very sensitive to the environment, including pH conditions, ionic strength effect, and the addition of analytes producing an aggregation phenomenon [29]. Therefore, tartrate acted

as a stabilizing agent and played a critical role in the capping formation of the monodispersed nanoparticles after the reducing process. The capping agent gives a negative surface charge, resulting in electrostatic repulsion and increasing nanoparticles' stability [8]. Surface chemistry stability depends on the surrounding nanoparticles' composition (molecule content, pH). A

complex environment can cause exchange phenomena during storage [9].

Size and Morphology Analysis of the Effect of Ascorbic Acid on the Synthesized Tartrate Capped AgNPs

TEM analysis aims to confirm the morphology of the produced nanoparticles. TEM images and the histogram of the produced tartrate capped AgNPs were presented in Fig. 2. The measurement of the particle size distributions along with the increasing concentration of L-ascorbic acid (25, 100, and 200 mM) were 27, 17, and 11 nm, respectively. A previous study stated increasing L-ascorbic acid concentration decreased particle size [20].

A small concentration of L-ascorbic acid as a reducing agent resulted in a partially remaining unchanged silver ion and produced the bigger particle size of nanoparticles as represented by the TEM image in Fig. 2(a). The higher concentration of L-ascorbic acid, the smaller particle size was achieved in the reducing process of Ag^+ into Ag^0 because the surface of produced AgNPs is adsorbed by oxidation products and inhibits nanoparticles from growing further resulting in ultrafine AgNPs. Therefore, as the concentration of L-ascorbic acid increased, the encapsulated Ag^+ decreased, resulting in smaller AgNPs (Fig. 2(b)).

While an excess concentration of L-ascorbic acid caused explosive nucleation, a complex compound was formed from the lone pair electrons of L-ascorbic acid's polar groups that occupy two sp orbitals of the silver ion. Then Ag^+ is reduced to Ag^0 inside the nanoscopic templates. In nanoscopic templates, large random nuclei will be formed by homogeneous nucleation, and small silver nanoparticles are easily assembled. The silver core will grow into primary nanoparticles and be aggregated into microspheres to reduce the total energy system. The formed nanoparticles produce defects in their structure to become sites for further deposition of silver. Crystal growth was affected by the oxidation byproduct of L-ascorbic acid on the AgNPs after the reduction process was finished. L-ascorbic acid is oxidized into its byproduct dehydroascorbic acid during this process. The structure of dehydroascorbic acid shows three carbonyl

groups (1,2,3-tricarbonyl) which are very electrophilic. The interaction between 3-carbonyl and 6-OH groups rapidly forms the hemiacetal species. Dehydroascorbic acid resulted in pricks on the microsphere's surface and finally evolved into rough surface structures [21]. Therefore, the influence of excess L-ascorbic acid concentration produced explosive nucleation with a rough surface, as confirmed by the TEM image in Fig. 2(c).

Proposed Mechanism

The results show that tartrate-capped AgNPs can be achieved by reducing Ag^+ by L-ascorbic acid and capping using disodium tartrate. L-ascorbic acid is easily soluble in water that acts as vinylogous carboxylic acid. A conjugated system is formed by the lactone ring carbonyl double bond, the hydroxyl group lone pair, and the electrons in the double bond [1]. L-ascorbic acid acts as a stable (electron+proton) donor in the redox system. It changed into the radical ion species called semidehydro[ascorbic acid] and was further converted into dehydroascorbic acid's final product. Dehydroascorbic acid and L-ascorbic acid constitute the redox system sufficient to reduce Ag^+ to Ag^0 (Fig. 3).

Reduction of Ag^+ into Ag^0 by L-ascorbic acid and capped with tartrate gives stable colloidal AgNPs. The oxidation of L-ascorbic acid is a typical spontaneous redox system [1]. The nucleation rate goes up due to the increased reactivity of the L-ascorbic acid in an alkaline solution. Interaction of tartrate with Ag^0 is critical to control nanoparticle formation and prevent further aggregation and agglomeration. The proposed mechanism of tartrate capped AgNPs formation is displayed in Fig. 4.

The Stability Study of the Effect of Ascorbic Acid on the Synthesized Tartrate Capped AgNPs

The stability of nanoparticles is one of the critical parameters to further applications [9]. The use of a capping agent can prevent the agglomeration of particles. The prepared tartrate capped AgNPs with the high L-ascorbic acid concentration of 25, 100, and 200 mM gave lower stability as indicated by decreasing

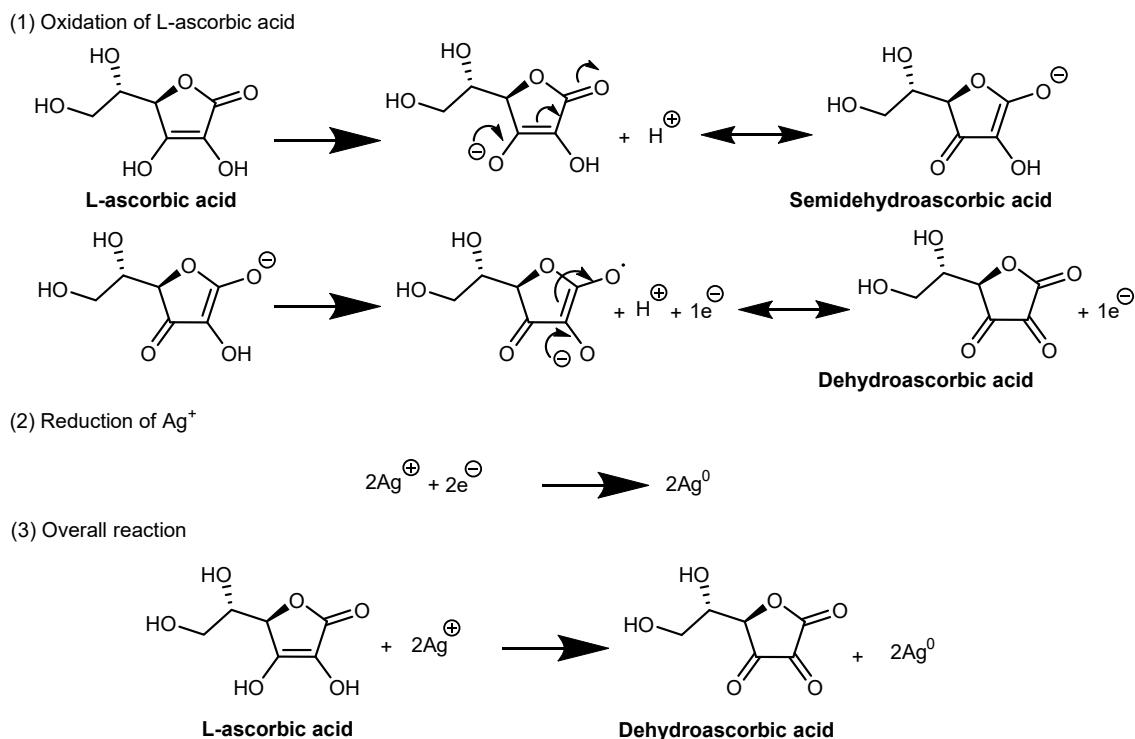


Fig 3. The reduction reaction for the formation of silver nanoparticles

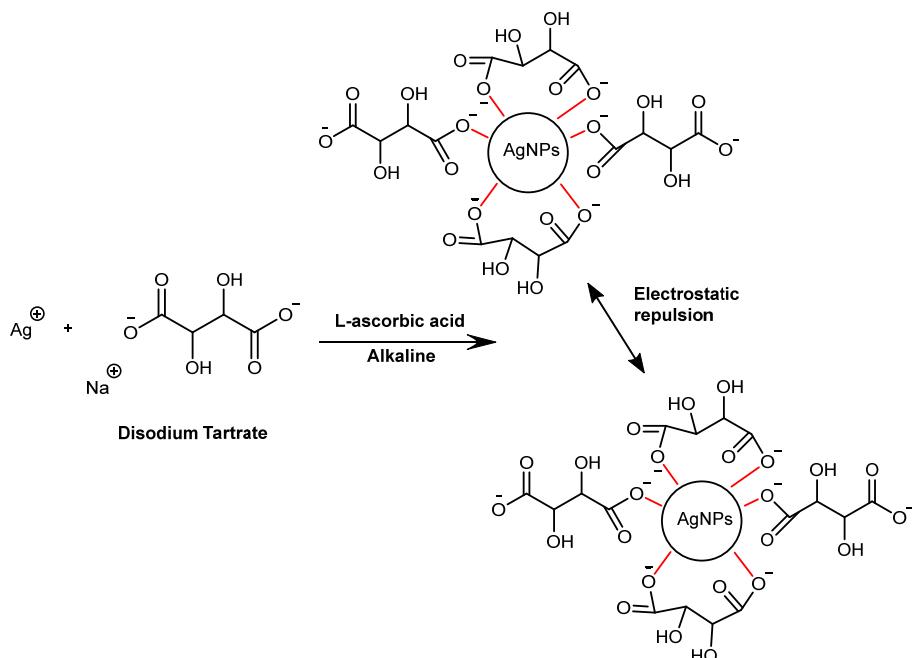


Fig 4. Proposed mechanism of tartrate capped AgNPs formation and stabilization

the zeta potential point of -33.5 , -20.8 , and -21.3 , respectively (Fig. 5). The stability of the product upon storage is shown in Fig. 6. The absorbance of the colloids decreased after one week with the order of L-ascorbic acid

concentrations of 25 , 100 , and 200 mM, which are 0.151 , 0.0105 , and 0.336 .

The results suggest that a low concentration of L-ascorbic acid (25 mM) has the highest zeta potential

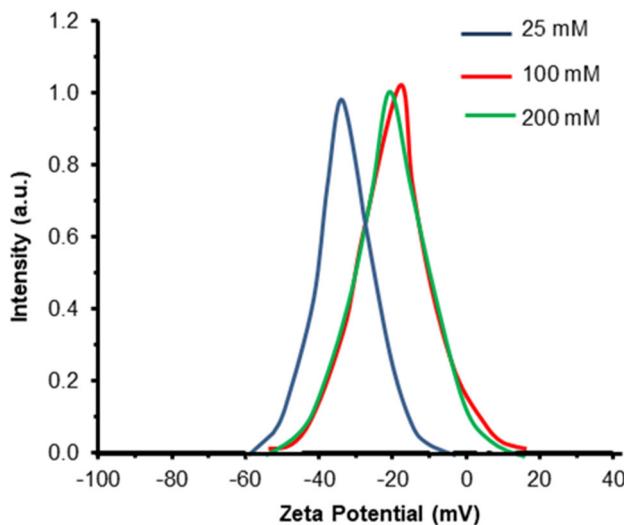


Fig 5. Zeta potential of tartrate capped AgNPs prepared in 25, 100, and 200 mM L-ascorbic acid

value, but it caused a faster decrease in absorbance than 100 mM. Meanwhile, the high concentration of L-ascorbic acid of 200 mM resulted in the most significant

reduction instability compared to the others. The zeta potential was similar for 100 mM. Thus, we may conclude that low or high levels of reducing agents resulted in low stability. Therefore, we should control the use of L-ascorbic acid concentration. The appropriate capping agent concentration can improve the produced nanoparticle stability.

UV-Visible spectra evaluated the stability of the produced colloidal AgNPs extended measurement during storage for AA 100 mM. Fig. 7 displays the UV-Vis spectra of the colloid measured from freshly prepared to 16 weeks. On day one, the peak absorbance was 1.1137, decreased to 0.9232 after 12 weeks, and decreased to 0.3411 after 16 weeks of storage. It means that the colloid is stable for up to 12 weeks. The lower the intensity, the broader the peak with the longer storage time. It indicated the reduced homogeneity of the particle size caused by the formation of larger particle sizes due to the nanoparticle aggregation and agglomeration to yield the

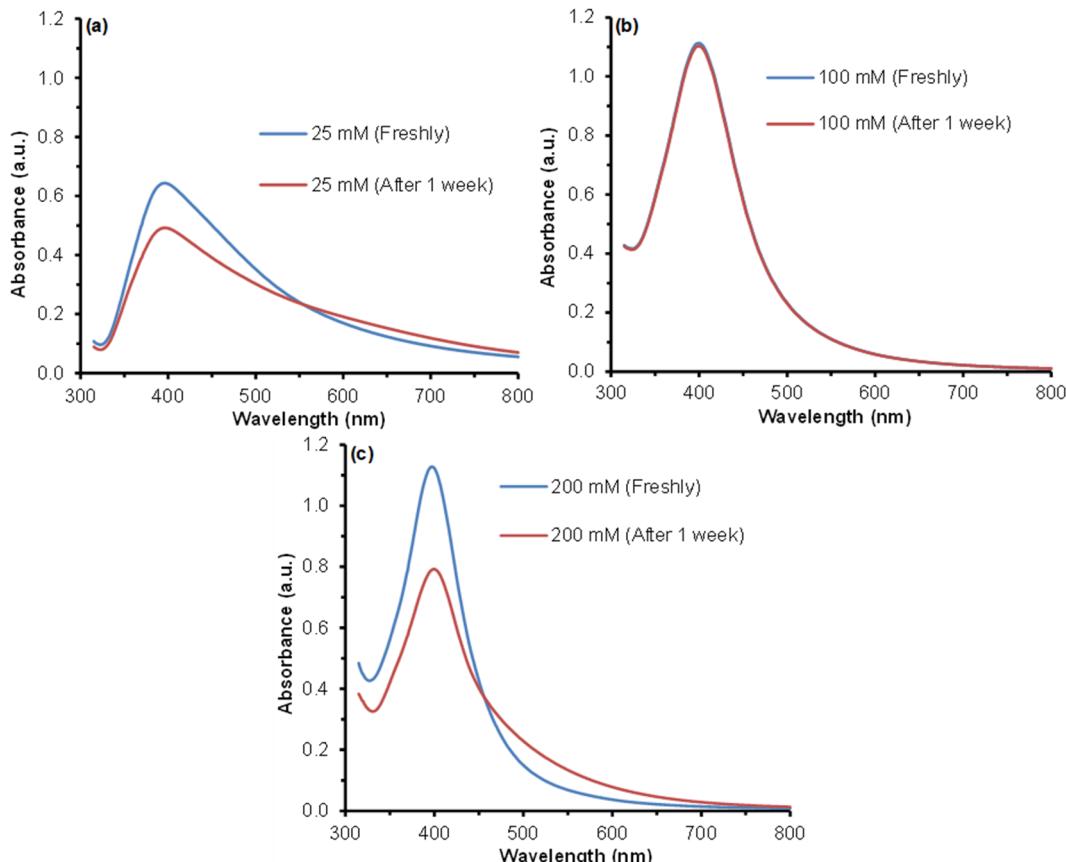


Fig 6. UV-Visible spectra of tartrate capped AgNPs by use of L-ascorbic acid (a) 25 mM, (b) 100 mM, and (c) 200 mM, freshly prepared and after one week

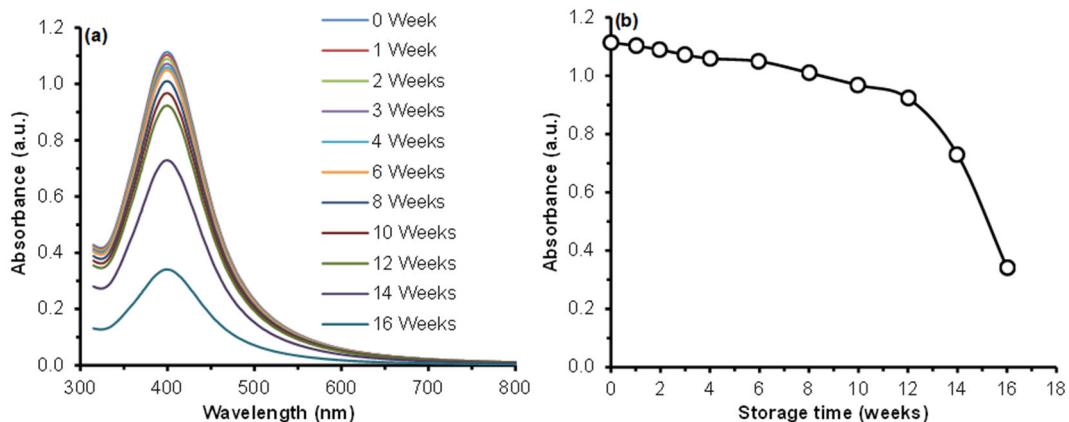


Fig 7. The absorbance change over the tartrate time capped AgNPs from week 1 to week 16

Table 1. Size distribution and zeta potential values of metal NPs in comparison with previous literature

Capping Agent	Reducing agent	Size distribution(nm)	Zeta potential (mV)	Reference
Chitosan capped AgNPs	ascorbic acid	200–700	+23.8 to +32.1	[30]
AA capped AuNPs	ascorbic acid	20–40	-4	[17]
PVA capped AgNPs	ascorbic acid	30	-10.40	[16]
Tartrate capped AgNPs	ascorbic acid	17	-20.8	This work

polydispersity of the colloid. The FWHM of the SPR absorbance peaks described the dispersity of nanoparticles, where a large FWHM value indicated the peak broadening due to increased polydispersity of nanoparticles [27].

The comparison between metal NPs size and stability reported earlier by L-ascorbic acid-reducing agents and this work is given in Table 1. The more positive or negative the zeta potential value, the better stability [13]. The produced tartrate capped colloidal AgNPs have relatively small particle sizes and good stability.

■ CONCLUSION

The concentration of ascorbic acid plays an important role in controlling the size, morphology, and stability of the produced tartrate capped AgNPs. Increasing L-ascorbic acid concentration from 25 mM to 100 mM and 200 mM decreased the particle size distributions from 27.29 to 16.87 and 10.72 nm, respectively. The zeta potential decreased from -33.5, -20.8, and -21.3 mV, lowering the absorbance peak after a week to 0.151, 0.0105, and 0.336, respectively. Reduction of silver nitrate by L-ascorbic acid is best performed at the concentration of 100 mM with a reaction time of around 30 min. The obtained colloids have good stability for the first 12 weeks. Based on our

result (1), the low concentration of L-ascorbic acid generated the bigger size and irregular shape of AgNPs. (2) As the concentration increases, the size of AgNPs were decreased with spherical shape and better uniformity due to the stabilizing effect. Meanwhile, the excess concentration resulted in explosive nucleation, generating the sea-urchin architecture. The less or excessive L-ascorbic acid concentration resulted in low stability. Therefore, the right concentration of L-ascorbic acid and appropriate capping agent can produce stable nanoparticles with homogeneous size and shape, essential for future applications.

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